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Refractomet Division

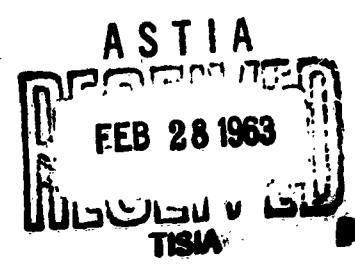
UNIVERSAL-CYCLOPS STEEL CORPORATION

Technical Report

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Bridgeville, Pennsylvania

ASC TR 7-786 (III)

ASC INTERIM REPORT 7-786 (III)
January, 1963

INFAB PROCESSING OF TZM SHEET

Contract AF33(657)-8495
Third Interim Technical
Engineering Report
15 August 1962 - 15 January 1963.

Phase III Report

Prepared By
F. R. Cortes

UNIVERSAL-CYCLOPS STEEL CORPORATION
REFRACTOMET DIVISION
BRIDGEVILLE, PENNSYLVANIA

The Phase III Program Production and Evaluation of Sheet Bar has been completed. Phase IV, Intermediate Rolling and Evaluation, is nearing completion.


BASIC INDUSTRY BRANCH
MANUFACTURING TECHNOLOGY LABORATORY
Directorate of Materials and Processes
Aeronautical Systems Division
United States Air Force
Wright-Patterson Air Force Base, Ohio

ABSTRACT
Third Interim Report

ASC INTERIM REPORT 7-786 (III)
January, 1963

INFAB PROCESSING OF TZM SHEET

Refractomet Division
Universal-Cyclops Steel Corporation



Seven extruded billets and two of four ingot sections were successfully impact forged to sheet bar and evaluated. Each of the nine sheet bar was cut into four sections and these were rolled to 0.125* intermediate gauge at temperatures from 2000°F to 3200°F. A total of 29 pieces of intermediate gauge strip were produced and partially evaluated for soundness and evidence of contamination. The Phase III program has been completed while completion of 0.125* mold out evaluation and pack rolling studies remain in the Phase IV program.

* in.



UNIVERSAL - CYCLOPS STEEL CORPORATION

BRIDGEVILLE, PENNSYLVANIA

REPLY TO:
BRIDGEVILLE PLANT
BRIDGEVILLE, PENNA.

February 22, 1963

Armed Services Technical Information
Center
Arlington Hall Station
Arlington 12, Virginia

Gentlemen:

Subject: InFab Processing of TZM Sheet
Contract No. AF33(657)-8495
Third Interim Technical Report

Enclosed is a copy of Interim Report No. 3 prepared under
United States Air Force, Manufacturing Technology Laboratory,
Contract AF33(657)-8495. This report covers the work on Phases
III and IV of this contract.

Any questions relative to the information contained herein may
be referred to the writer.

Very truly yours,

UNIVERSAL-CYCLOPS STEEL CORPORATION



L. M. Bianchi
Technical Manager
Refractomet Division

LMB:mlb

Enclosure

ASC TR 7-786 (II)

ASC INTERIM REPORT 7-786 (III)
January, 1963

INFAB PROCESSING OF TZM SHEET

Contract AF33(657)-8495
Third Interim Technical
Engineering Report
15 August 1962 - 15 January 1963

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F. R. Cortes

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FOREWORD

This Interim Technical Progress Report covers work performed under Contract AF33(657)-8495 from 15 August 1962 to 15 January 1963. It is published for technical information only and does not necessarily represent the recommendations, conclusions, or approval of the Air Force.

This contract with the Refractomet Division of Universal-Cyclops Steel Corporation, Bridgeville, Pennsylvania was initiated under ASC Aeronautical System Division, Project 7-786, "InFab Processing of TZM Sheet." It was administered under the direction of Mr. Hugh L. Black, Project Engineer, Basic Industry Branch, Manufacturing Technology Laboratory, Wright-Patterson Air Force Base, Ohio. F. R. Cortes of the Development Group, Refractomet Division, Universal-Cyclops Steel Corporation was the engineer in charge.

Since the nature of this work is of interest to so many fields of endeavor, your comments are solicited as to the potential utilization of the material produced under this contract. In this manner, it is felt that a full realization of the resultant material produced will be accomplished.

PUBLICATION REVIEW

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I. Introduction

This program was designed to evaluate the potential of the InFab facility for the production of TZM alloy sheet. The evaluation consists of the following six phase program:

- Phase I Literature Survey
- Phase II Ingot Production and Evaluation
- Phase III Production of Sheet Bar
- Phase IV Intermediate Breakdown
- Phase V Production of Evaluation Sheets
- Phase VI Production of Sheets by Best Techniques Developed

During the last report period Phase III was completed and the Phase IV program was begun. This report covers all the requirements of the Phase III program and work to date on Phase IV. The Phase IV rolling program was followed as previously outlined except for a reduction in the rolling temperature range from 2400°F.-3850°F. to 2000°F.-3200°F.

Evaluation of Phase IV intermediate gauge is nearing completion and will allow choice of Phase V sheet rolling variables prior to resumption of InFab operations in March. At that time pack rolling evaluation will be initiated which will complete the Phase IV program.

II. Phase III - Sheet Bar Forging and Evaluation

A. Forging of Extruded and Cast Billets

Eleven forging billets, 7 extruded and 4 as-cast, were forged according to the modified Phase III forging schedule Figure I, and the detailed forging parameters are listed in Table I. Prior to preparing extruded

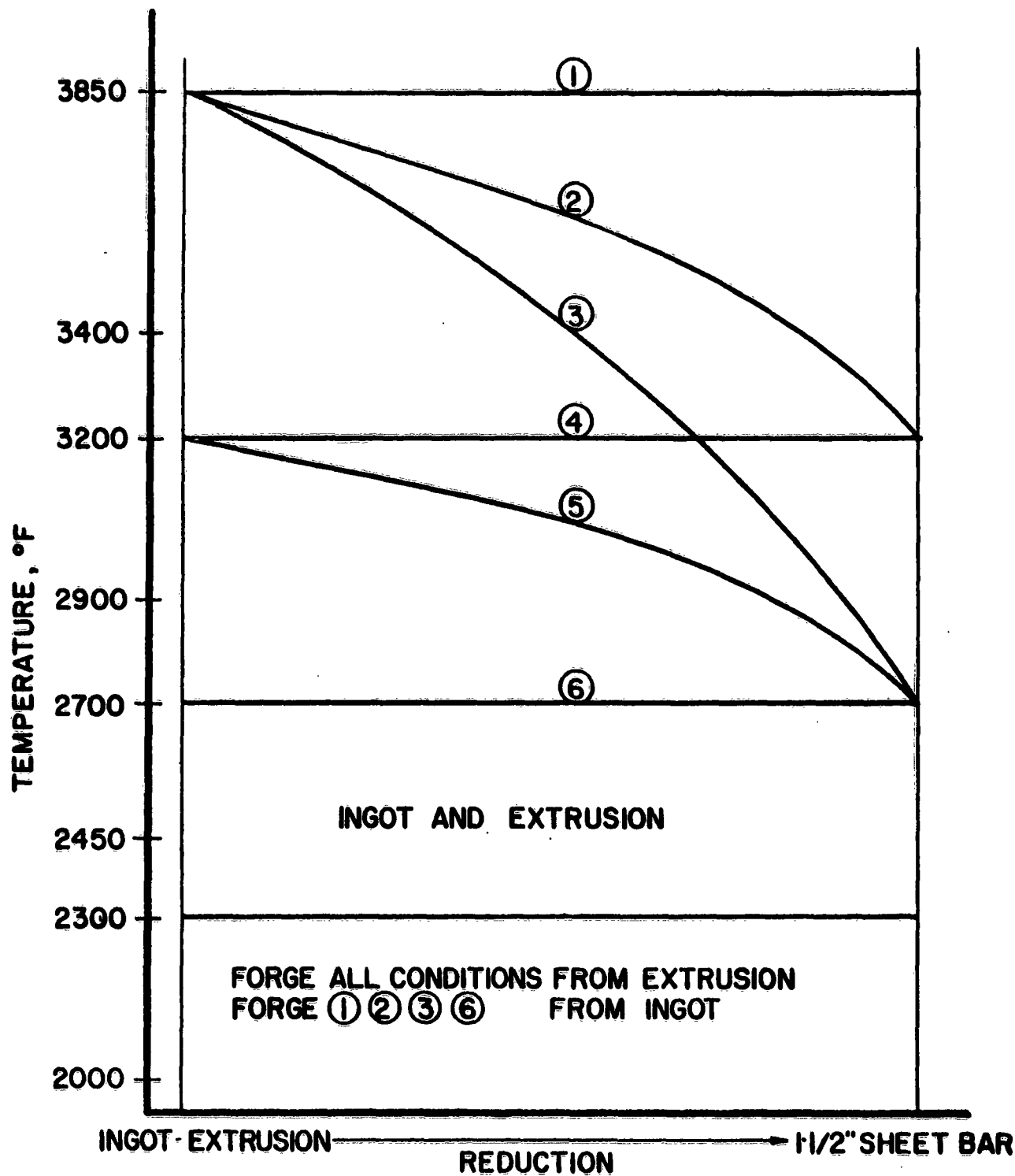


FIGURE 1

INFAB HOT-WORK PROGRAM—PHASE III—FORGING OF SHEET BAR

TABLE I
FORGING PARAMETERS

[illegible]

forging billets, the extrusions had been hydrogen annealed one hour at 2800°F. Since this did not produce a fully recrystallized structure, extruded billets (1098B-3 and 1098B-4), which were scheduled for forging below 3200°F., were given a ten minute anneal at 2950°F. in the InFab forge furnace prior to forging to effect full recrystallization.

Initial breakdown of cast billets to approximately 3-3/4" diameter was accomplished by V-die forging, providing a working stress distribution which minimized the possibility of center burst. Final forging was then performed with flat faced dies. Extruded billets were forged to both intermediate 3-3/4" square and final 1-1/2" sheet bar on flat faced forging dies. For billets scheduled for forging entirely at one temperature, Figure 1, the minimum temperature in both the intermediate and final forging operations were maintained at from 200°F. to 300°F. below the starting temperature. For billets scheduled for forging over a broad temperature range, the initial forging to intermediate size was begun at the temperatures shown in Figure 1 while the finish forging operation was initiated at the minimum temperatures shown and allowed to drop another 300°F. to 400°F. The actual forging temperatures listed in Table I were for the most part in good agreement with the aims.

Forging of the seven extruded billets presented no major difficulties. However, the material was noticeably tougher to forge than lower carbon TZM previously forged on Contract NOas 59-6142-c. The seven billets were forged to a sheet

bar cross-section of about 4" x 1-1/2" representing reductions of from 50 to 58%. Figure 2 shows a typical sheet bar produced from extruded billet after having been cut into sections for evaluation and rolling.

As expected, the four cast billets were much less fabricable than extruded billets and the lowest initial forging temperature proved most detrimental. Billet 1099D forged at 2700°F., Figure 3 showed severe surface cracks after the first forging blow and as forging continued the piece was destroyed by center burst. This failure was intergranular and is attributable to the continuous boundary carbides in the cast structure. Forging of Billet 1099B, 3850°F. start and a 3400°F. minimum, proceeded fairly well to intermediate round except for a 5" section which broke off the bottom of the billet. However on finish forging with flat dies, severe edge splitting penetrated to the center of the sheet bar, Figure 4. This failure is attributed to the fact that the intermediate hot forged 3-3/4" did not differ much from a cast structure and since final forging on flat dies above 3300°F. did not transmit any cold work to the piece or allow carbides to move out of the grain boundaries, slight edge cracking was easily propagated with continued forging.

Billets 1099A and 1099C, hot forged to intermediate size at 3850°F. but finished at temperatures below 2800°F., forged with minor difficulties, Figures 5 and 6, but sufficient material was produced for rolling in Phase IV.

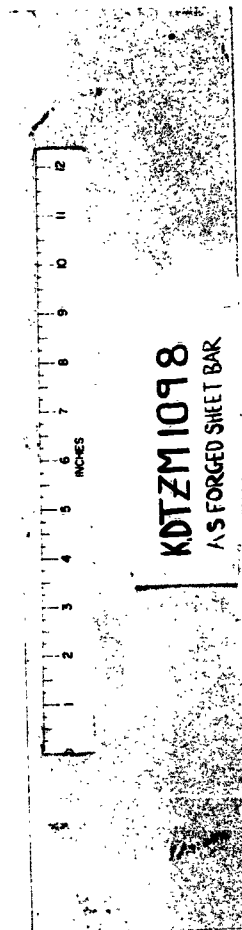
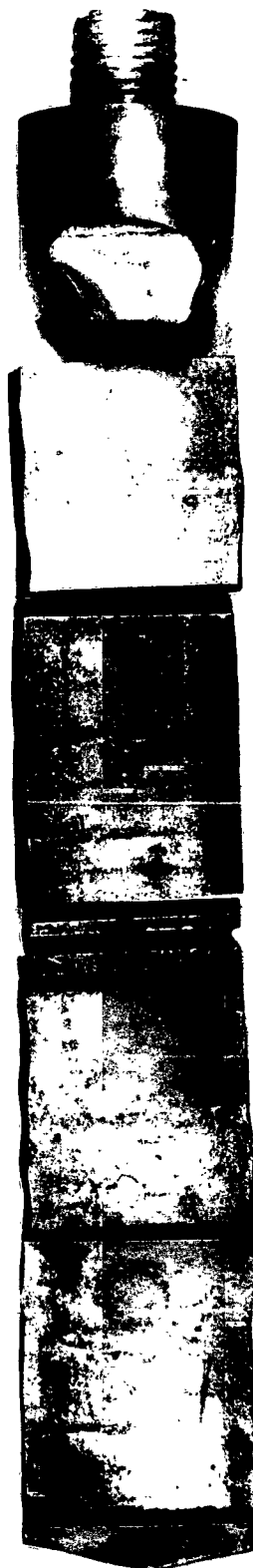


FIGURE 2 - SHEET BAR FORGED FROM 4" DIAMETER EXTRUSION BILLET

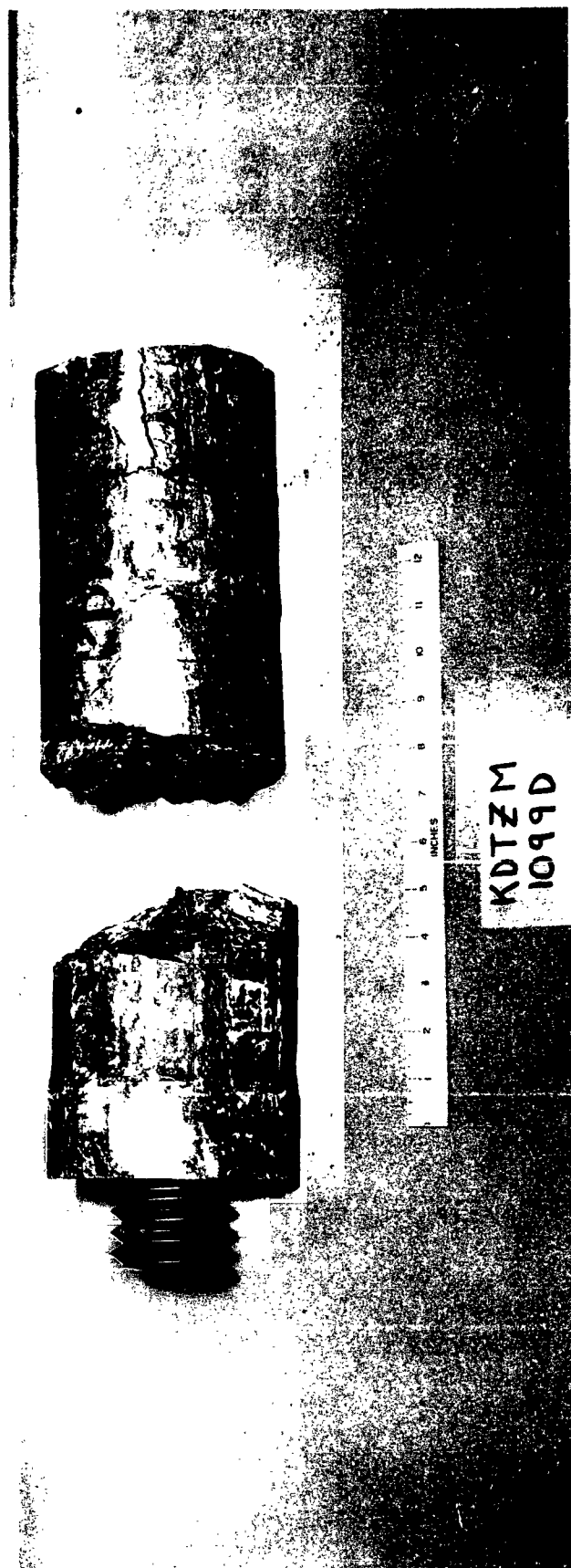


FIGURE 3 - AS-CAST BILLET 1099D FORGED AT 2700°F.

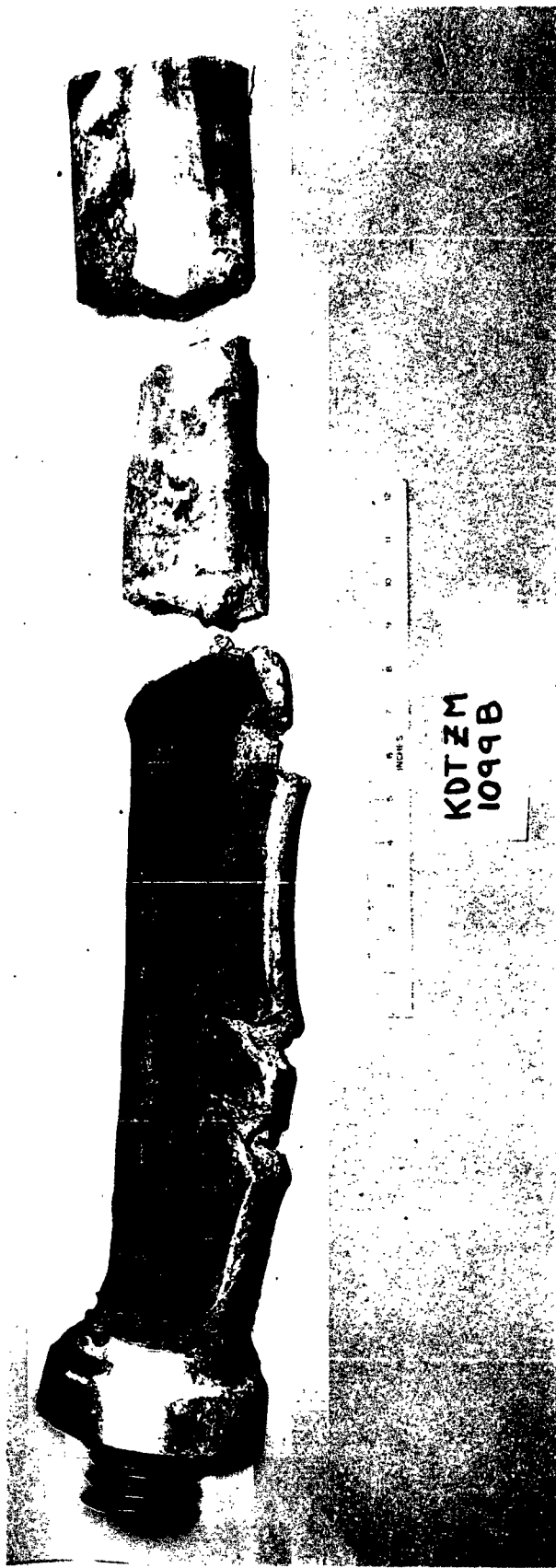


FIGURE 4 - AS-CAST BILLET 1099B FORGED BETWEEN 3870°F. AND 3300°F.



FIGURE 5 - AS-CAST BILLET 1099C FORGED BETWEEN 3850°F. AND 2770°F.

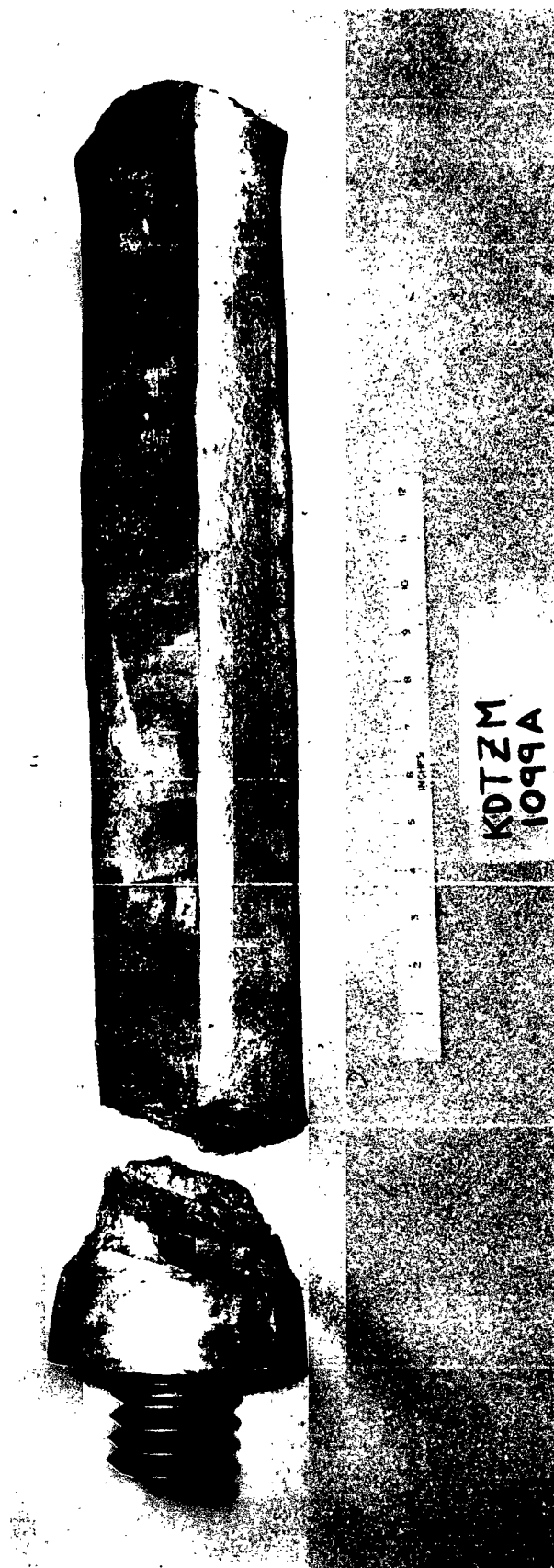


FIGURE 6 - AS-CAST BILLET 1099A FORGED BETWEEN 3870°F. AND 2400°F.

A 6" piece was severed off of Billet 1099C in the initial forging operation and minor edge or side cracks occurred in final forging. Of the as-cast billets, 1099A finished to the best appearance, Figure 6, as not even minor edge cracks occurred in final forging at temperatures as low as 2400°F. However, the sheet bar did sever from the holder bar section before final thickness was obtained.

B. Sheet Bar Quality

1. Ultrasonic Inspection

No defects were found in as-forged sheet bar by ultrasonic inspection techniques. Cracks appearing in the macro of sheet bar 1098B3, Figure 9, apparently occurred during preparation of the macro as no sonic indication was found in the sheet bar and little difficulty was encountered rolling sections from this sheet bar.

2. Macrostructures

Transverse slices for macrostructural examination were cut from the center of each sheet bar. Hardnesses were taken on three slices at edge and center locations and specimens for heat treatment response were cut from the center of each slice.

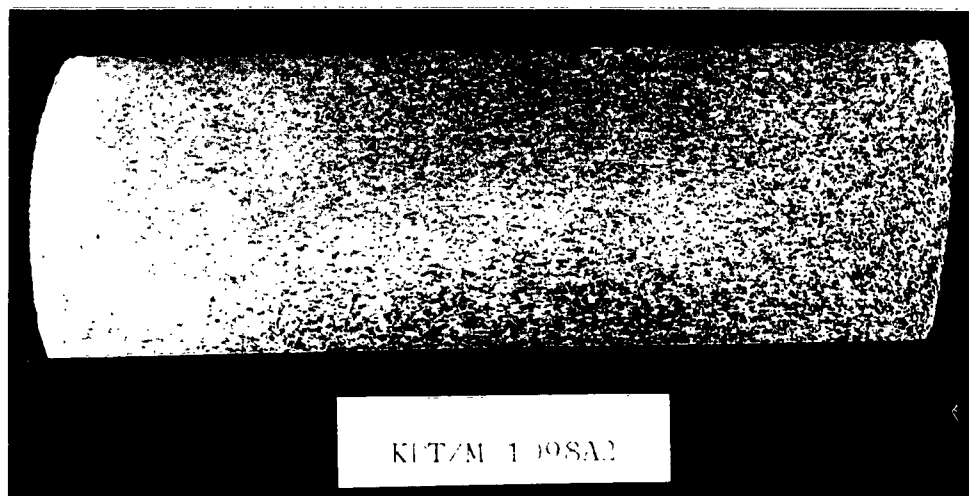
Macros of the three extruded billets forged at 3850°F. and finished at progressively lower temperatures are shown in Figure 7. Billet 1098A-1 exhibits a fully hot forged structure with a coarse grain diameter (>ASTM 1) varying from about .3 to .7 mm. Billets

FINISH
FORGING
TEMPERATURE

3350°F.



2770°F.



2350°F.

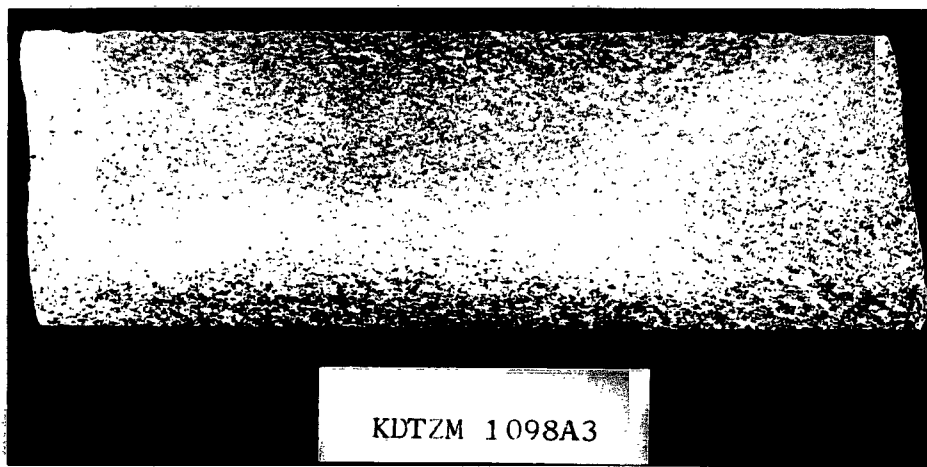


FIGURE 7 - MACROSTRUCTURE OF SHEET BARS FORGED FROM
4" EXTRUSION AT AN INITIAL TEMPERATURE OF 3850°F. AND
FINISHED AT PROGRESSIVELY LOWER TEMPERATURES

1098A2 and 1098A3 developed the same hot forged grain size during the initial hot forging but finish forging at progressively lower temperatures increased grain size and transmitted increasing amounts of hot cold work as evident in the macros.

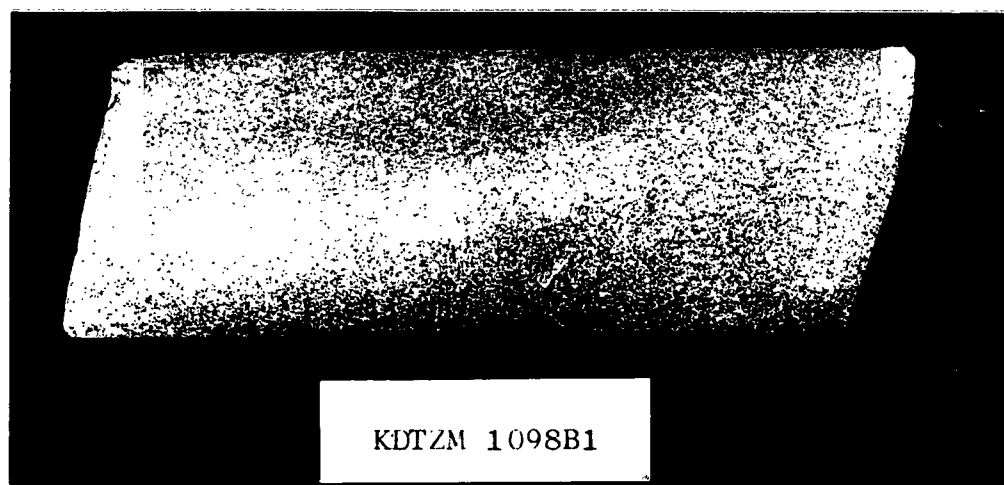
The macro of extruded Billet 1098B1, forged entirely at 3200°F., reveals a very uniform hot forged structure with a grain size of about ASTM 2.5, Figure 8.

Macros of Billets 1098B2, B3 and B4, forged at 3200°F., 2700°F. and 2300°F. respectively, exhibit non-uniformly wrought structures, Figure 9. Billet 1098B4 appeared more heavily worked than the other two and somewhat more uniform.

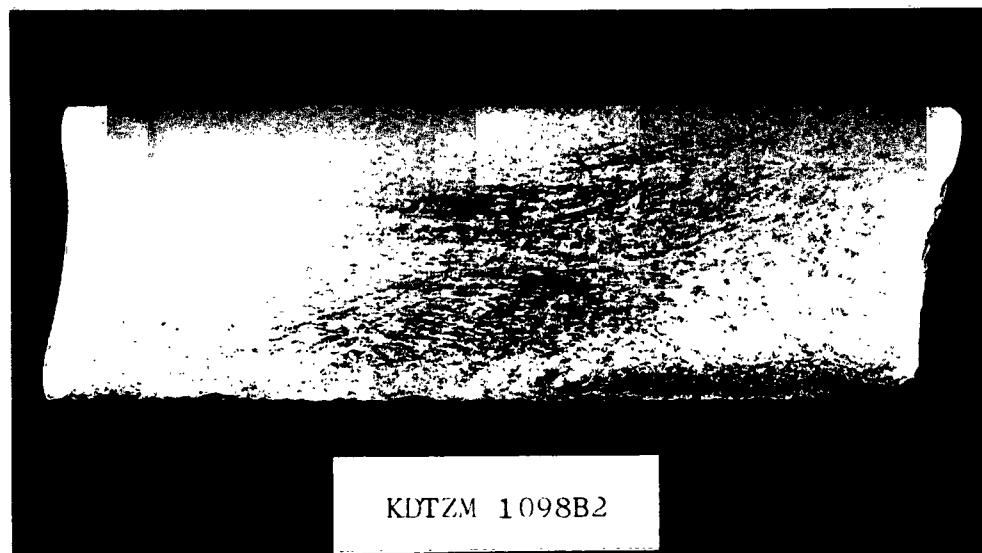
Macros of sheet bar forged from as-cast Billets 1099A and 1099C are shown in Figure 10. It is evident that the cast billets result in the more coarse sheet bar structures. In addition, a contaminated surface is revealed in the macro of these two sheet bars.

3. Microstructures

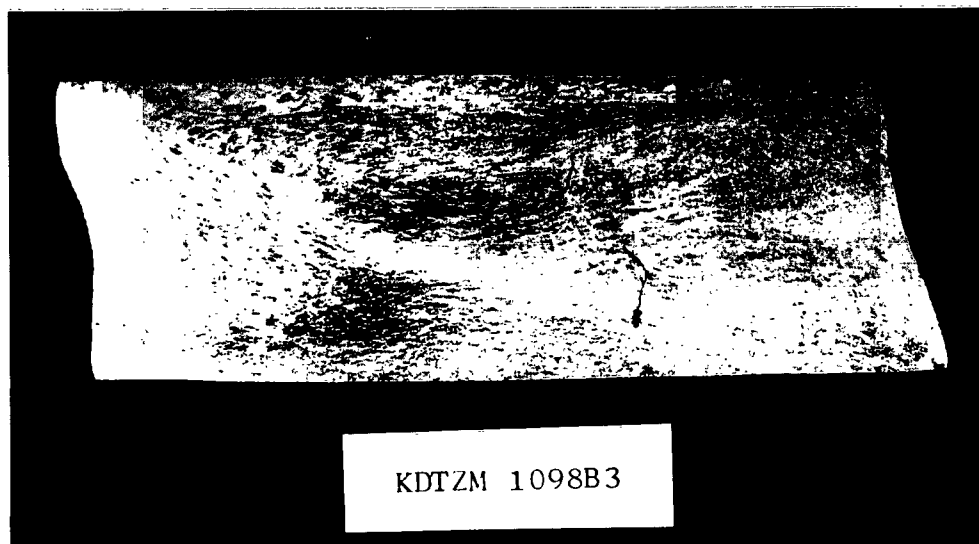
Microstructures of sheet bar, Figures 11 through 15, were taken to reveal the type of surface contamination rather than the typical as-forged structure. However, the structures shown are fairly typical of the whole sheet bar except for Billets 1098A2 (Figure 11) and 1098B3 (Figure 13) which exhibited more heavily worked centers.



**FIGURE 8 - MACROSTRUCTURE OF SHEET BAR FORGED
FROM 4" EXTRUSION AT 3200°F.**



KDTZM 1098B2



KDTZM 1098B3



KDTZM 1098B4

FIGURE 9 - MACROSTRUCTURE OF SHEET BAR HOT-COLD
FORGED FROM 4" DIAMETER RECRYSTALLIZED EXTRUSION

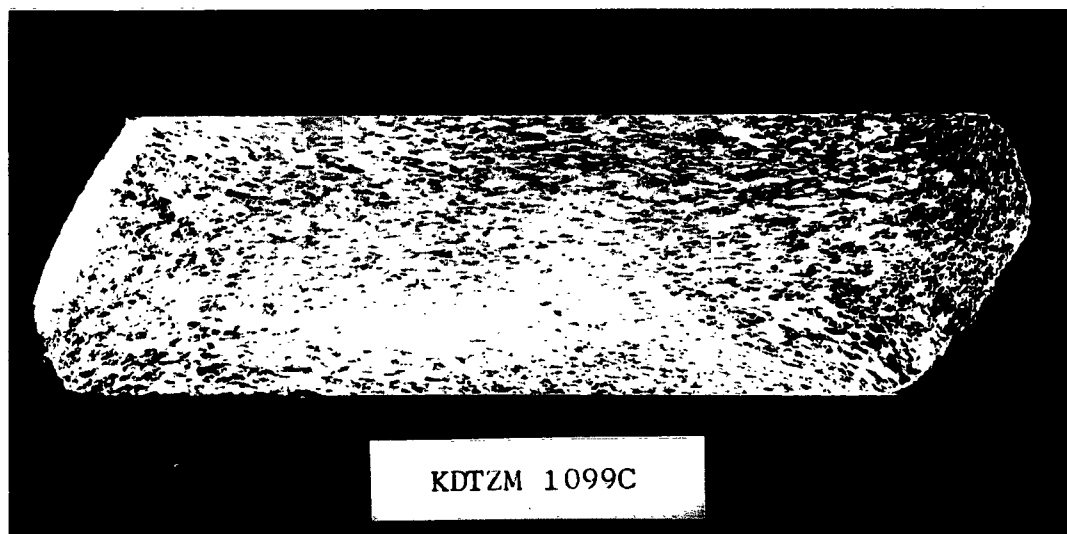
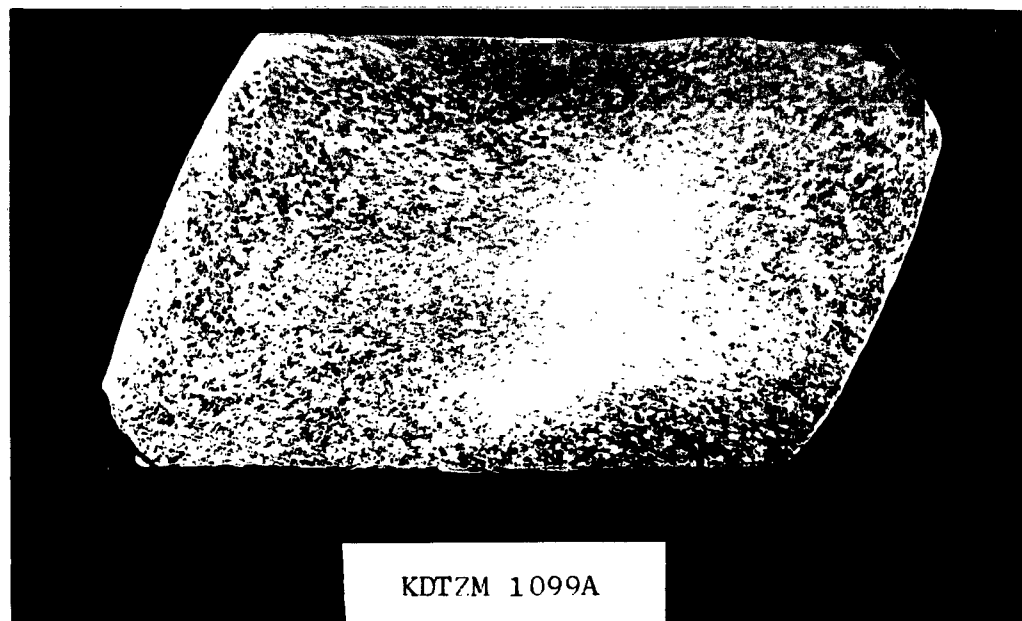
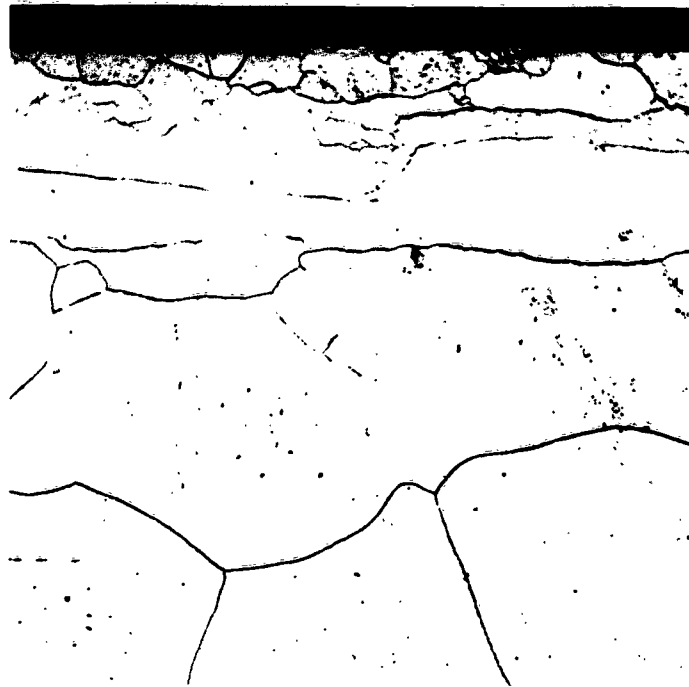
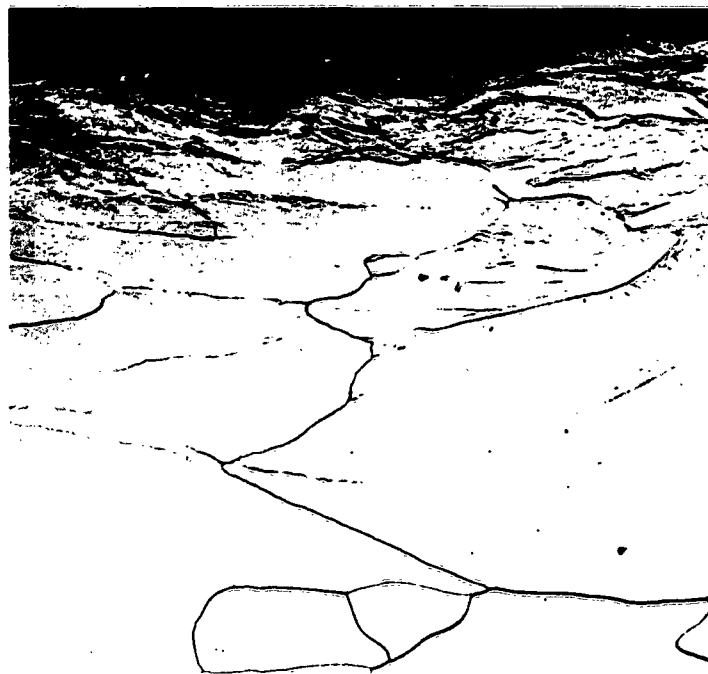


FIGURE 10 - MACROSTRUCTURE OF SHEET BARS HOT
FORGED FROM 6" DIAMETER INGOT SECTIONS

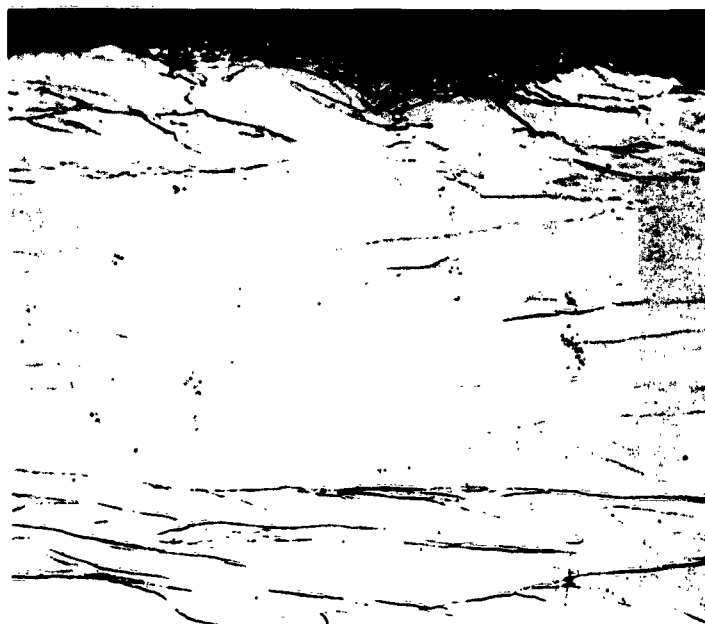


R12665 SURFACE AT MIDDLE OF S.B. 200X
1098A1 FORGED AT 3810°F. START, FINISHED AT 3350°F.

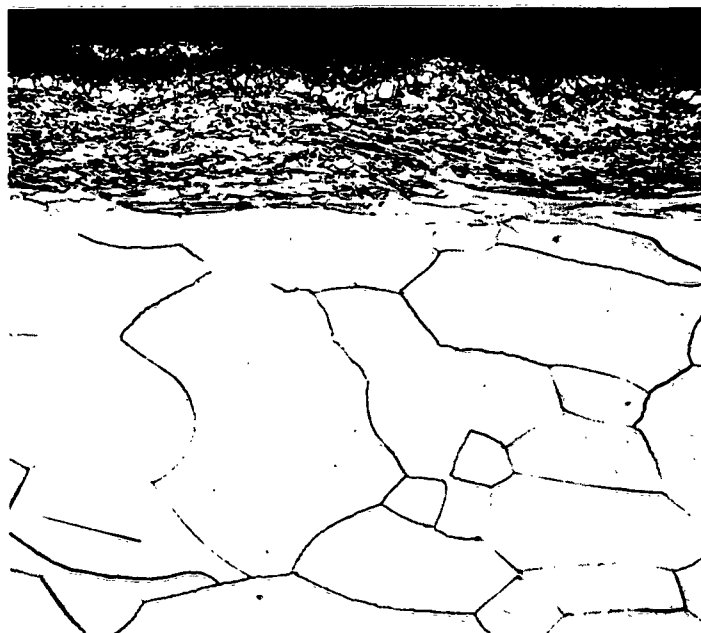


R12666 SURFACE AT MIDDLE OF S.B. 200X
1098A2 FORGED AT 3720°F. START, FINISHED AT 2770°F.

FIGURE 11 - MICROSTRUCTURES OF SHEET BAR HOT FORGED
FROM 4" DIAMETER EXTRUSION

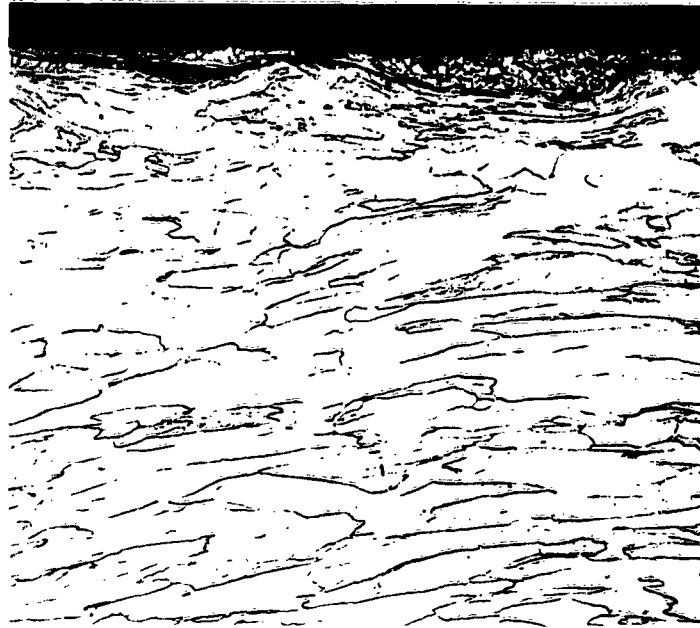


R12667 SURFACE AT MIDDLE OF S.B. 200X
1098A3 FORGED AT 3810°F. START, FINISHED AT 2350°F.

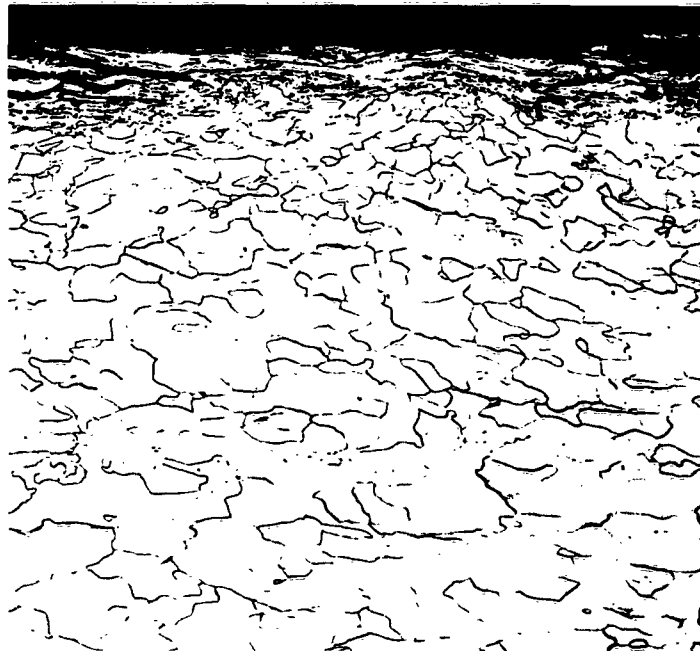


R12668 SURFACE AT EDGE OF S.B. 200X
1098B1 FORGED AT 3200°F. START, FINISHED AT 2950°F.

FIGURE 12 - MICROSTRUCTURE OF SHEET BAR HOT FORGED
FROM 4" DIAMETER EXTRUSION



R12669 SURFACE AT MIDDLE OF S.B. 200X
1098B2 FORGED AT 3120°F. START, FINISHED AT 2300°F.



R12670 SURFACE AT MIDDLE OF S.B. 200X
1098B3 FORGED AT 2600°F. START, FINISHED AT 2310°F.

FIGURE 13 - MICROSTRUCTURE OF SHEET BAR HOT-COLD
FORGED FROM 4" DIAMETER EXTRUSION



R12672

SURFACE AT MIDDLE OF S.B.

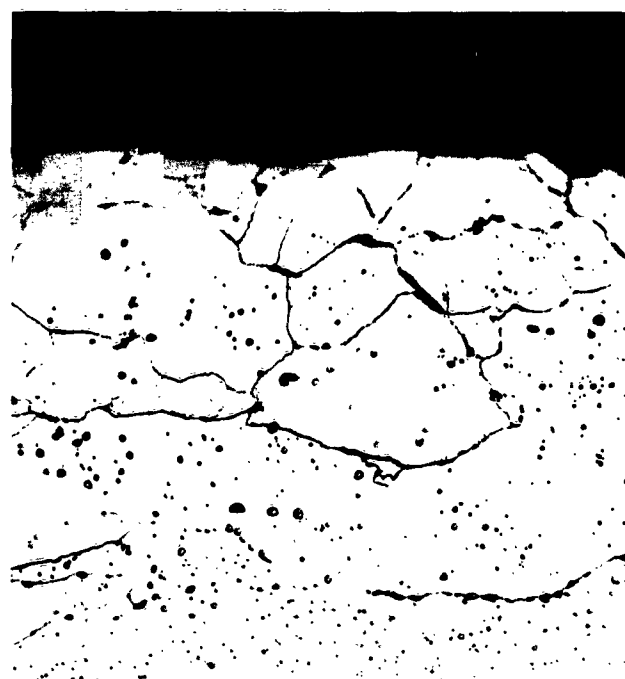
200X

1098B4 FORGED AT 2320°F. START, FINISHED AT 1950°F.

FIGURE 14 - MICROSTRUCTURE OF SHEET BAR HOT-COLD
FORGED FROM 4" DIAMETER EXTRUSION

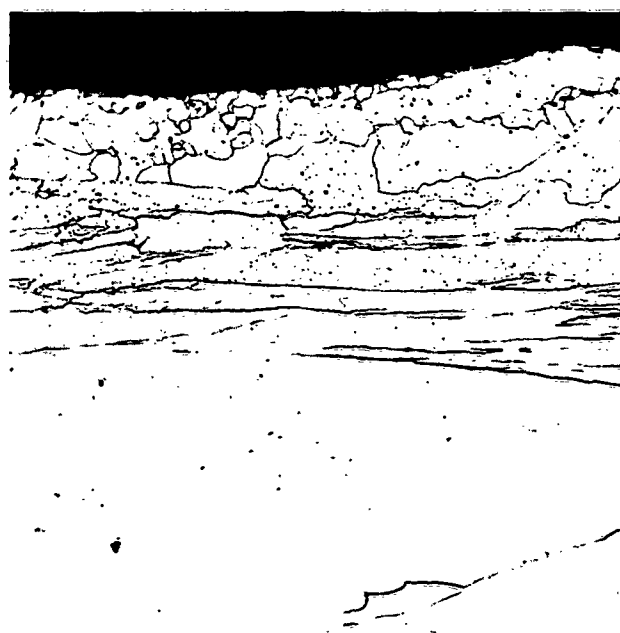


R12684 200X
SURFACE AT EDGE OF S.B.

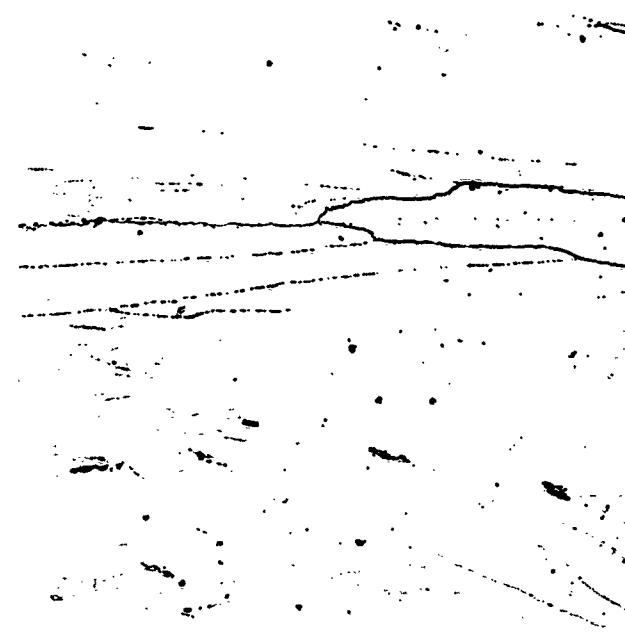


R12685 500X
SURFACE AT EDGE OF S.B.

1099A FORGED AT 3870°F. START, FINISHED AT 3300°F.



R12673 200X
SURFACE AT EDGE OF S.B.



R12674 500X
CENTER OF S.B.

1099C FORGED AT 3850°F. START, FINISHED AT 2770°F.

FIGURE 15 - MICROSTRUCTURE OF SHEET BAR HOT
FORGED FROM 6" DIAMETER INGOT

a. Contamination

Contamination in molybdenum alloys hot worked in air has been detected metallographically by a difference in recrystallization temperatures between the contaminated layer and the base metal. Interstitial contamination in air has been found to increase recrystallization temperature resulting in wrought surface layers on recrystallized or hot worked structures. In recent work on the Navy's Molybdenum Sheet Rolling Program as well as in this work, iron contamination has been evident in InFab during fabrication at temperatures much above the melting point of the forging dies and roll surfaces. The form taken by this iron contamination has not been conclusively determined. However where surface iron is found to be high, a second type of surface layer has been noted, consisting of a fine recrystallized structure with a dark etching constituent primarily in the boundaries. When both types of surface layers occur together, the wrought layer is generally found beneath the recrystallized surface.

Figures 11 through 15 show that recrystallized surface layers appear on all as-forged sheet bar except the two, 1098B3 and 1098B4, forged at the lowest temperatures. The recrystallized layers, on the sheet bars forged from cast billets, Figure 15, are particularly coarse and contain a second phase both at the boundaries and in the matrix.

As yet, this phase is unidentified. Wrought layers are evident on sheet bars 1098B1, B2, B3 and both sheet bars 1099A and 1099C forged from cast billet. It is impossible, however, to determine accurate contamination depths metallographically since contamination can be present below the visibly affected surface layers. For example, Billet 1098A1, Figure 11, exhibits a light recrystallized layer but no wrought layer since the very high forging temperature of 3810°F. was sufficient to offset any increase in recrystallization temperature caused by interstitial contamination. Furthermore, the recrystallized layer, attributed to iron contamination, exhibits relatively coarse grains compared to similar layers on sheet bar forged at lower temperatures such 1098A3 and 1098B1, Figure 12.

b. Carbide Distribution

Carbide distribution in as-forged sheet bar showed significant variation with forging practice as the following table describes. Magnifications of at least 500X were required to determine the carbide morphology.

| Sheet Bar | Forging Temperature | | Carbide Distribution |
|-----------|---------------------|---------|--|
| | Start | Finish | |
| Extruded | | | |
| 1098A1 | 3810°F. | 3350°F. | Fine Carbides in Matrix & Boundaries |
| 1098A2 | 3720°F. | 2770°F. | Fine Carbides in Matrix & Boundaries & Carbide Stringers |
| 1098A3 | 3810°F. | 2350°F. | Fine Carbides in Matrix & Boundaries & Carbide Stringers |

| Sheet Bar | Start | Finish | Carbide Distribution |
|-----------|---------|---------|--|
| 1098B1 | 3200°F. | 2950°F. | Continuous Carbide at Boundaries— Matrix Clean |
| 1098B2 | 3120°F. | 2300°F. | Very Fine Carbides in Matrix |
| 1098B3 | 2600°F. | 2310°F. | Few Carbides Evident in Worked Structure |
| 1098B4 | 2320°F. | 1950°F. | Few Carbides Evident in Worked Structure |
| Cast | | | |
| 1099A | 3870°F. | 3300°F. | Continuous Boundary Carbide—Some Gross Carbides |
| 1099C | 3850°F. | 2770°F. | Continuous Boundary Carbide—Some Gross Carbides and Carbide Stringers |

Sheet Bar 1098B1, which was hot forged in a narrow temperature range at around 3200°F., exhibited only boundary carbides. Maintaining 3200°F. until forging was complete prevented precipitation of titanium carbide (TiC) and only molybdenum carbide (Mo_2C) formed at temperature was evident at the boundaries. Extruded and cast billets hot forged at 3700°F. to 3800°F. but finished at temperatures in the range from 2300°F. to 3100°F. exhibited both boundary and matrix carbides. Reheats in this temperature range provided the opportunity for matrix precipitation which remained fine during the finish forging. Gross molybdenum carbides and carbide stringers were also present in sheet bar forged from cast billets since initial forging temperatures were in the stability range for Mo_2C , thus maintaining the coarse carbides from the ingot. The stringers result from gross carbides in the ingot boundaries. Carbides in

Billets 1098B2, B3 and B4 were very fine due to the very low forging temperatures and heavy cold reductions. A greater amount of TiC would be present in these billets.

4. Hardness

Vickers hardness (10 kg load) was obtained on sheet bar macros at six locations as listed in Table II.

Fully hot forged sheet bars 1098A1 and 1098B1 exhibit the lowest average hardness levels, 244 and 249 respectively, and as expected the peak as-forged hardness, 288, occurred in Billet 1098B4, forged at the lowest temperature. A high hardness of 280 was also developed in Billet 1098A3 even though its initial forging temperature was 3810°F. This high hardness is attributed to the fact that only two reheats were used in forging this billet. The uniformity of the macrostructure of Billet 1098B1 is again confirmed by the uniformity of the as-forged hardness which showed a variation with location of only 8 points from 245 to 253. For the most part, the hardness data show good uniformity for forged sheet bar as a spread of from 20 to 30 points is not excessive considering the influence of edge and surface cooling affects. Non-uniformity is greatest in two of the extruded billets; one, forged at the very highest temperature (1098A1) and one forged at the lowest temperature (1098B4).

TABLE II
Hardness of Impact Forged TZM Sheet Bar

| Billet | Forging Temperatures °F. | | Location: | Edge | Mid.Rad. | Center | Spread | Range | Avg. |
|--------|-----------------------------|--------|-----------|------|----------|--------|--------|---------|------|
| | Start | Finish | | | | | | | |
| 1098A1 | 3810 | 3350 | Surface | 268 | 245 | 247 | | | |
| | | | Center | 260 | 230 | 221 | 47 | 221-268 | 244 |
| 1098A2 | 3720 | 2770 | Surface | 372 | 272 | 274 | | | |
| | | | Center | 270 | 254 | 258 | 20 | 254-274 | 267 |
| 1098A3 | 3810 | 2350 | Surface | 276 | 270 | 276 | | | |
| | | | Center | 283 | 292 | 285 | 20 | 272-292 | 280 |
| 1098B1 | 3200 | 2950 | Surface | 249 | 245 | 251 | | | |
| | | | Center | 250 | 253 | 245 | 8 | 245-253 | 249 |
| 1098B2 | 3120 | 2300 | Surface | 262 | 238 | 243 | | | |
| | | | Center | 254 | 266 | 254 | 28 | 238-266 | 253 |
| 1098B3 | 2600 | 2310 | Surface | 264 | 247 | 256 | | | |
| | | | Center | 264 | 283 | 266 | 36 | 247-283 | 263 |
| 1098B4 | 2320 | 1950 | Surface | 266 | 283 | 274 | | | |
| | | | Center | 274 | 302 | 322 | 56 | 266-322 | 288 |
| 1099A | 3870 | 2400 | Surface | 297 | 266 | 279 | | | |
| | | | Center | 283 | 279 | 281 | 31 | 266-297 | 281 |
| 1099C | 3850 | 2770 | Surface | 256 | 256 | 254 | | | |
| | | | Center | 281 | 268 | 260 | 27 | 254-281 | 262 |

5, Recrystallization

The heat treatment response of sheet bar forged from extruded and cast billets is shown in Figures 16 and 17. Of the two extruded and forged billets, 1098A1 and 1098B1, exhibiting fully hot forged structures, the one forged at the lower temperature, 1098B1 at 3200°F., maintained the highest hardness after one hour at temperatures up to 3000°F. The only misconstructural change noted in these two hot forged billets with temperatures up to 3000°F. was a change in carbide morphology. Above 2800°F., fine carbides appeared in the matrix and boundary carbides become less evident indicating conversion of molybdenum carbides to the more stable titanium carbides in this temperature range.

Considering sheet bar containing hot cold work, billets 1098A2 and 1098A3 exhibit the highest recrystallization temperatures since they had been only partially forged below the true hot working temperature, Table I. The hardness change with temperature of 1098A3 follows that of fully hot forged 1098B1 very closely. Microstructures of both 1098A2 and 1098A3 exhibited only trace recrystallization after one hour at 3000°F. The three billets forged almost entirely below the hot work range, 1098B2, B3 and B4 exhibit the greater softening with temperature as recrystallized hardness levels of 195 to 205 are reached at 2900°F. 1098B4, in particular, softened most rapidly and was fully recrystallized at about 2800°F.

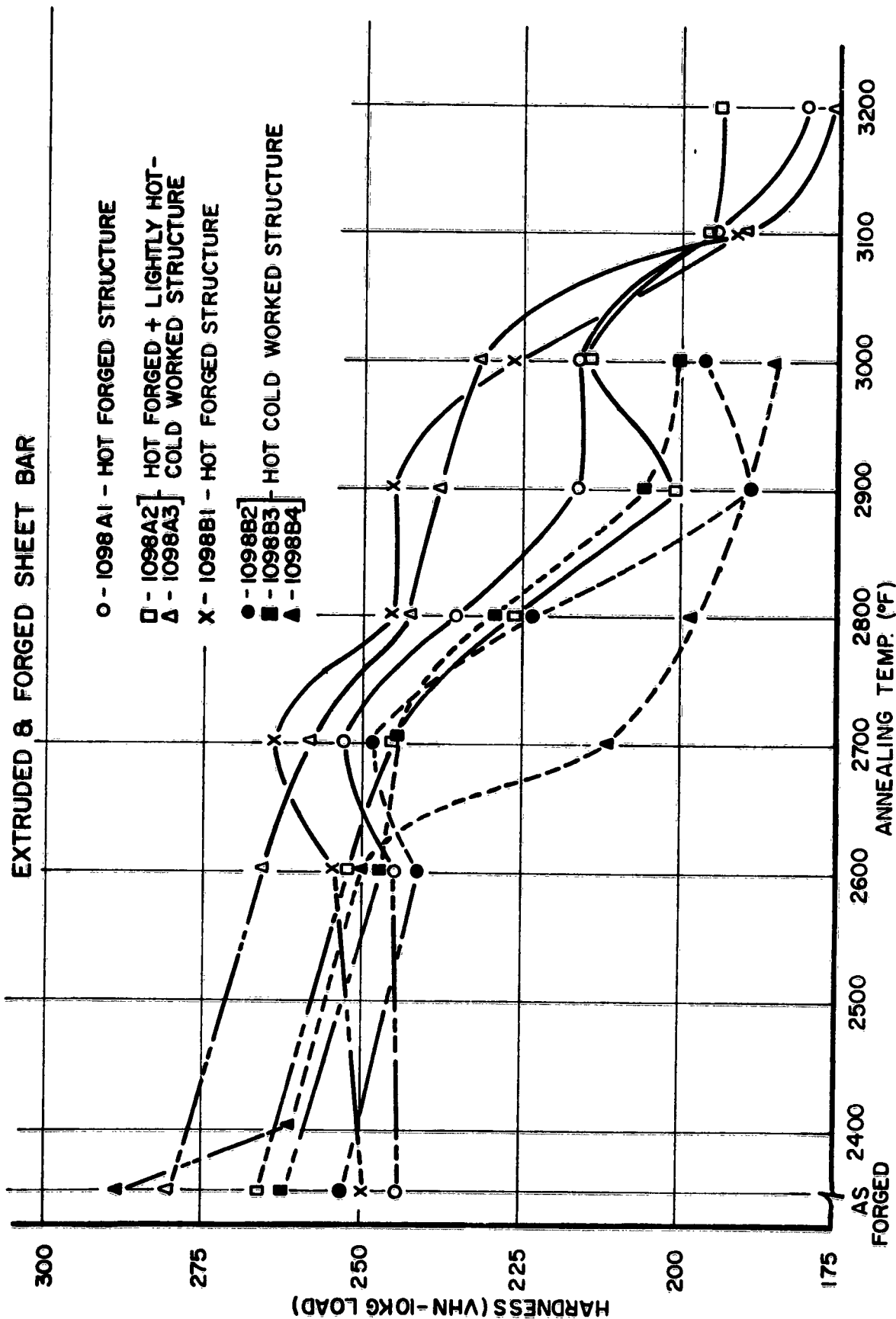


FIGURE 16
EFFECT OF ANNEALING TEMPERATURE ON THE HARDNESS
OF EXTRUDED AND FORGED SHEET BAR

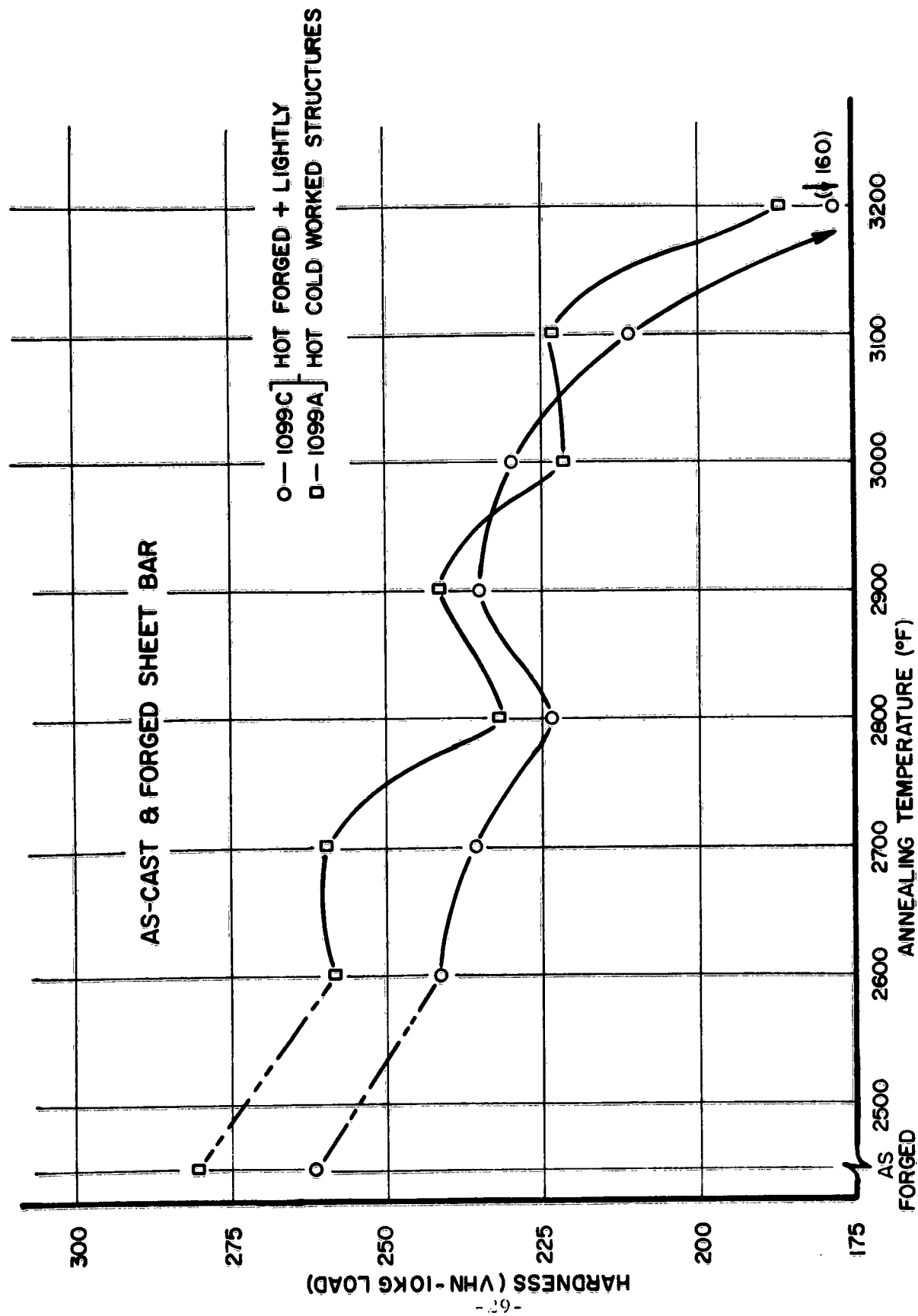


FIGURE 17
EFFECT OF ANNEALING TEMPERATURE ON THE HARDNESS OF
CAST & FORGED SHEET BAR

Softening curves for sheet bar forged from cast billets (Figure 17) again reveal the influence of a larger percentage of hot work than cold work as recrystallized hardness levels are attained at annealing temperatures above 3100°F.

The following table lists the temperature required for 50% recrystallization in each sheet bar as estimated by microstructural examination.

| Sheet Bar | Temperature for 50% Recrystallization In One Hour (°F.) |
|-----------|--|
| Extruded | |
| 1098A1 | Hot Forged |
| 1098A2 | 3150 |
| 1098A3 | 3150 |
| 1098B1 | Hot Forged |
| 1098B2 | 2825 |
| 1098B3 | 2800 |
| 1098B4 | 2750 |
| Cast | |
| 1099A | 3000 |
| 1099C | 3150 |

III. Phase IV - Intermediate Breakdown and Evaluation

A. Rolling of Sheet Bar Sections to 0.125" Mold Out

According to the modified Phase IV program, each sheet bar was cut into four 4" to 5" long sections for cross-rolling to intermediate gauge at 2000°F., 2400°F., 2800°F. and 3200°F. Prior to rolling, each section was annealed in InFab for ten minutes at 2950° (except pieces for rolling at 3200°F.), and conditioned by belt grinding to remove discoloration or contamination incurred during forging or annealing.

Tables III and IV list 34 sheet bar sections and the parameters which describe the rolling to the nominal 0.125" mold out. Rolling to intermediate gauge at 2000°F., 2400°F. and 3200°F. was accomplished in from 9 to 12 passes with one or two passes per reheat. In each case reductions of from 17 to 20% were applied on the first and second passes and total reductions to finished gauge ranged from 90 to 92%. After rolling approximately three-fourths of the sheet bar sections, a mill bearing block was broken and the remaining sheet bar sections for rolling at 2800°F. were then finish rolled at much lighter reductions requiring about 25 passes.

From the 34 sheet bar sections rolled, 29 pieces of mold out were produced while five sections alligatored on the first or second pass. As shown in Tables III and IV, all five "alligatored" sections were from a sheet bar forging practice which produced a hot-worked or at most a lightly hot-cold worked structure. Furthermore, three of the five failures were rolled at the lowest temperature 2000°F., one at 2400°F. and one at 2800°F.; therefore, all five received a ten minute anneal at 2950°F. prior to rolling. (Only sheet bar rolled at 3200°F. were not annealed.) However, due to the forging practice, these sections would be least affected by this anneal. Intergranular failure was evident in the fractures of the "alligatored" sections indicating that the boundary carbides in the hot forged sheet bar inhibited low temperature fabricability. At higher temperatures (3200°F.) the carbide network at the grain boundaries would tend to redistribute thereby increasing fabricability of coarse hot worked structures. In future rolling of hot forged sheet bar at relatively low temperatures, use of a longer time high temperature anneal, at least one hour at 3000°F. or above, should increase low temperature fabricability.

TABLE III

| Billet Number | Aim Forging Practice | Cond. Sheet Bar Size | Sheet Bar Section Code | Rolling Temp. °F. | No. of Passes | No. of Reheats | Mold Out Produced |
|------------------|------------------------------|------------------------------------|------------------------------|-------------------------|------------------|-------------------|-------------------------|
| KDTM | | | | | | | |
| 1098A1 | Init/3850/Rehe/3850/Min/3500 | 4-3/4" x 1-1/2" x 4-1/8" x 11# | T-1 | 3200 | 9 | 6 | 5-5/8" x .132 x 14-1/2" |
| | | 4-3/4" x 1-5/8" x 4" x 12# | T-2 | 2800 | 2 | 2 | Alligatoring—No Product |
| | | 4-3/4" x 1-1/2" x 4-1/8" x 11# | T-3 | 2400 | 11 | 10 | 5-1/8" x .122 x 26-1/2" |
| | | 4-1/2" x 1-3/8" x 4" x 9# | T-4 | 2000 | 1 | 1 | Alligatoring—No Product |
| 1098A2 | Init/3850/Rehe/3200/Min/2800 | 5" x 1-5/8" x 4-1/2" x 12-1/2# | T-5 | 3200 | 13 | 7 | 5-3/4" x .138 x 41" |
| | | 5" x 1-1/2" x 4-5/8" x 14# | T-6 | 2800 | 26 | 7 | 5-3/8" x .119 x 33-3/4" |
| | | 4-3/4" x 1-1/2" x 4-1/2" x 12-1/2# | T-7 | 2400 | 1 | 1 | Alligatoring—No Product |
| | | 5" x 1-1/2" x 4-1/4" x 12# | T-8 | 2000 | 13 | 10 | 5-1/4" x .112 x 47" |
| 1098A3 | Init/3850/Rehe/2700/Min/2400 | 4-3/4" x 1-1/2" x 4-5/8" x 13# | T-9 | 3200 | 10 | 7 | 5-3/4" x .143 x 35-3/4" |
| | | 5-3/8" x 1-1/2" x 4-1/2" x 13# | T-10 | 2800 | 26 | 8 | 5-3/4" x .126 x 35-1/4" |
| | | 4-1/2" x 1-1/2" x 4-3/4" x 12-1/2# | T-11 | 2400 | 10 | 10 | 5" x .125 x 44-1/2" |
| | | 4-1/2" x 1-1/2" x 4-5/8" x 13# | T-12 | 2000 | 11 | 8 | 5" x .129 x 21-1/2" |
| 1098B1 | Init/3200/Rehe/3200/Min/3000 | 5-1/4" x 1-1/2" x 4-1/2" x 12-3/4# | T-13 | 3200 | 9 | 4 | 5-5/8" x .167 x 16-1/2" |
| | | 5-3/4" x 1-1/2" x 4-1/8" x 13-1/2# | T-14 | 2800 | 26 | 8 | 6-1/4" x .123 x 42-1/2" |
| | | 5-1/4" x 1-1/2" x 4-1/4" x 12-1/2# | T-15 | 2400 | 11 | 10 | 5-5/8" x .119 x 10" |
| | | 5-1/4" x 1-1/2" x 4-1/4" x 12# | T-16 | 2000 | 2 | 1 | Alligatoring—No Product |
| 1098B2 | Init/3200/Rehe/2700/Min/2300 | 5-1/8" x 1-1/2" x 4-5/8" x 15# | T-17 | 3200 | 9 | 5 | 5-1/2" x .152 x 25-1/2" |
| | | 5-1/2" x 1-1/2" x 4-3/4" x 16# | T-18 | 2800 | 25 | 7 | 6" x .120 x 50" |
| | | 5" x 1-1/2" x 4-5/8" x 15# | T-19 | 2400 | 11 | 10 | 5-1/8" x .125 x 46-1/2" |
| | | 5-1/4" x 1-1/2" x 4-5/8" x 15# | T-20 | 2000 | 12 | 9 | 5-1/8" x .125 x 44-1/2" |

TABLE IV

| Billet Number | Aim Forging Practice | Cond. Sheet Bar Size | Sheet Bar Code | Rolling Temp. °F. | No. of Passes | No. of Reheats | Usable Mold Out Produced |
|----------------------|------------------------------|------------------------------------|----------------|-------------------|---------------|----------------|--------------------------|
| KDITM | | | | | | | |
| 1098B3 | Init/2700/Rehe/2700/Min/2400 | 4-5/8" x 1-1/2" x 4-1/8" x 11# | T-21 | 3200 | 9 | 4 | 5-1/4" x .164 x 7" |
| | | 4-5/8" x 1-1/2" x 4-1/4" x 11-1/2# | T-22 | 2800 | 26 | 9 | 5-1/4" x .108 x 49-1/2" |
| | | 4-5/16" x 1-1/2" x 4-1/8" x 11# | T-23 | 2400 | 9 | 9 | 4-3/4" x .115 x 43" |
| | | 4-5/16" x 1-1/2" x 4-3/16" x -- | T-24 | 2000 | 7 | 7 | 4-7/8" x .116 x 44-1/2" |
| KDITM | | | | | | | |
| 1098B4 | Init/2300/Rehe/2300/Min/2000 | 5" x 1-1/2" x 4-1/2" x 12# | T-25 | 3200 | 9 | 4 | 5-1/4" x .150 x 13" |
| | | 4-3/4" x 1-1/2" x 4-1/2" x 11-1/2# | T-26 | 2800 | 25 | 7 | 5-1/4" x .125 x 41-1/4" |
| | | 3-7/8" x 1-1/2" x 4-1/4" x 10# | T-27 | 2400 | 9 | 9 | 4-3/8" x .117 x 45" |
| | | 4" x 1-1/2" x 4-1/2" x -- | T-28 | 2000 | 9 | 9 | 4-5/8" x .119 x 50-1/4" |
| As-Cast KDITM | | | | | | | |
| 1099A | Init/3850/Rehe/2700/Min/2400 | Eliminate—No Material | T-33 | 3200 | -- | -- | -- |
| | | 7" x 2-1/2" x 3-1/2" x 23-1/2# | T-34 | 2800 | 37 | 9 | 7-3/4" x .117 x 32-1/4" |
| | | 6-1/4" x 2-1/4" x 4" x 21# | T-35 | 2400 | 26 | 6 | 6-5/8" x .133 x 31" |
| KDITM | | | | | | | |
| 1099C | Init/3850/Rehe/3200/Min/2800 | Eliminate—No Material | T-36 | 2000 | 1 | 1 | Alligatore—No Product |
| | | 4-1/4" x 1-1/2" x 3-1/4" x 8# | T-37 | 3200 | -- | -- | -- |
| | | 4-3/4" x 1-1/2" x 3-7/8" x 10# | T-38 | 2800 | 25 | 7 | 4-3/4" x .127 x 25-1/2" |
| | | 5" x 1-3/8" x 4-1/2" x 13-1/2# | T-39 | 2400 | 24 | 5 | 5-1/2" x .123 x 32" |
| | | | T-40 | 2000 | 11 | 10 | 5-1/4" x .116 x 48" |

B. Evaluation of Intermediate Gauge

Figures 18 through 21 show as-rolled mold out grouped according to rolling temperatures. Close examination of each figure reveals the deterioration of mold out surfaces as rolling temperature increased. Mold out rolled at 2800°F. (Figure 20) exhibits a very coarse surface and, in addition, strip rolled at 3200°F. (Figure 21) exhibits the effects of roll surface seizing noted by localized surface de-lamination.

1. Ultrasonic Inspection

Ultrasonic inspection revealed up to 6" of unsound or laminated material on the end of some strips as well as up to 1/2" of laminated material along the side. The strip shown in Figures 18 through 21 are chalk marked to show the extent of unsound material. Also in some cases only short lengths of mold out were produced since the mold out was cut in half prior to finish rolling to minimize problems associated with handling very long narrow pieces at very high temperatures (above 2800°F.).

2. Contamination

Mold out samples in both the as-rolled and flash annealed conditions (five minutes @ 2800°F.) were examined for evidence of surface contamination and the results listed in Table V. Visible contamination on mold out rolled at 3200°F. and 2800°F. was difficult to measure because of their very coarse or uneven surfaces. No worked layer was visible on mold out rolled at 3200°F.



FIGURE 18 - NOMINAL 0.125" MOLD OUT
PRODUCED FROM SHEET BAR ROLLED AT 2000°F.

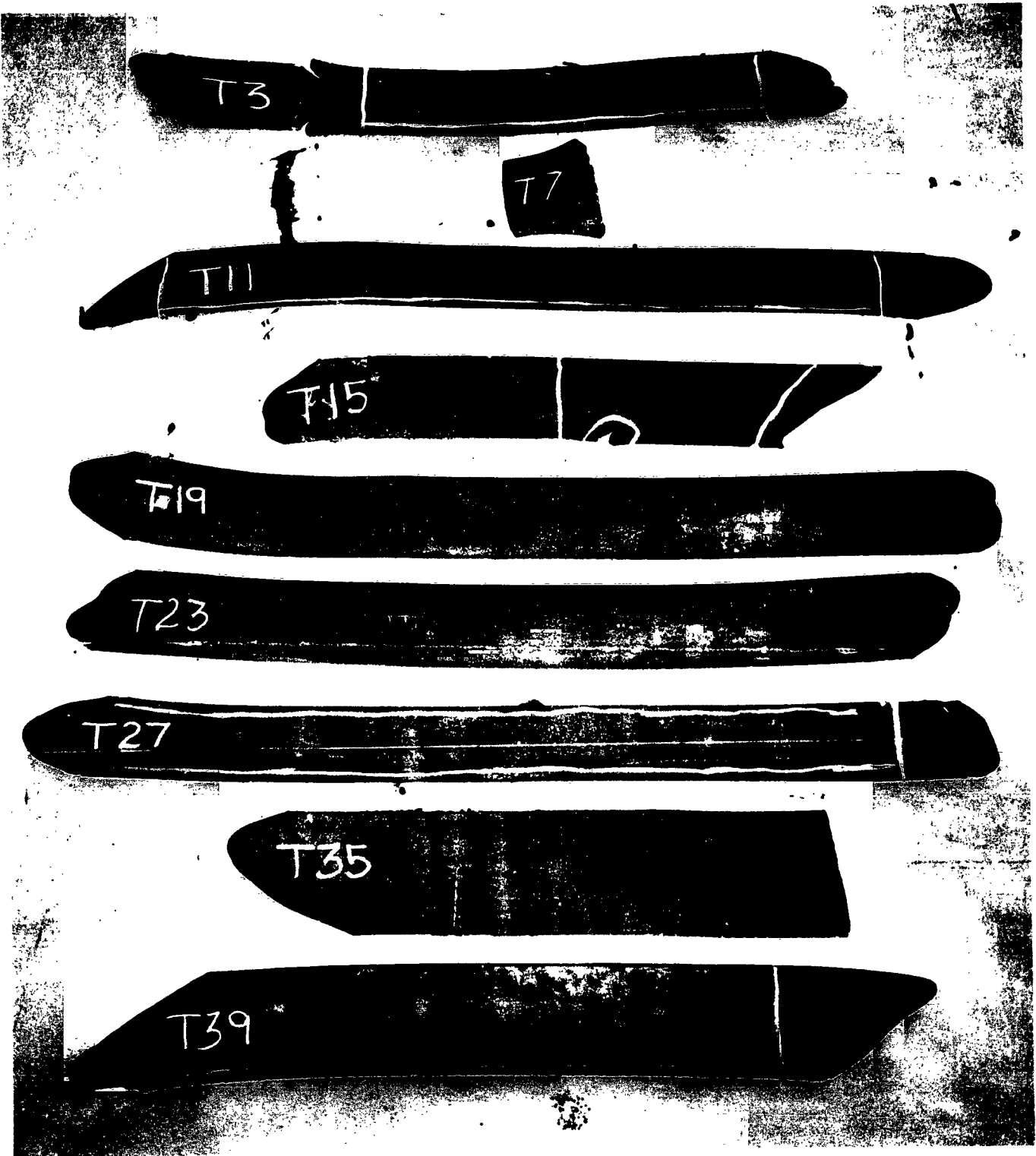


FIGURE 19 - NOMINAL 0.125" MOLD OUT
PRODUCED FROM SHEET BAR ROLLED AT 2400°F.

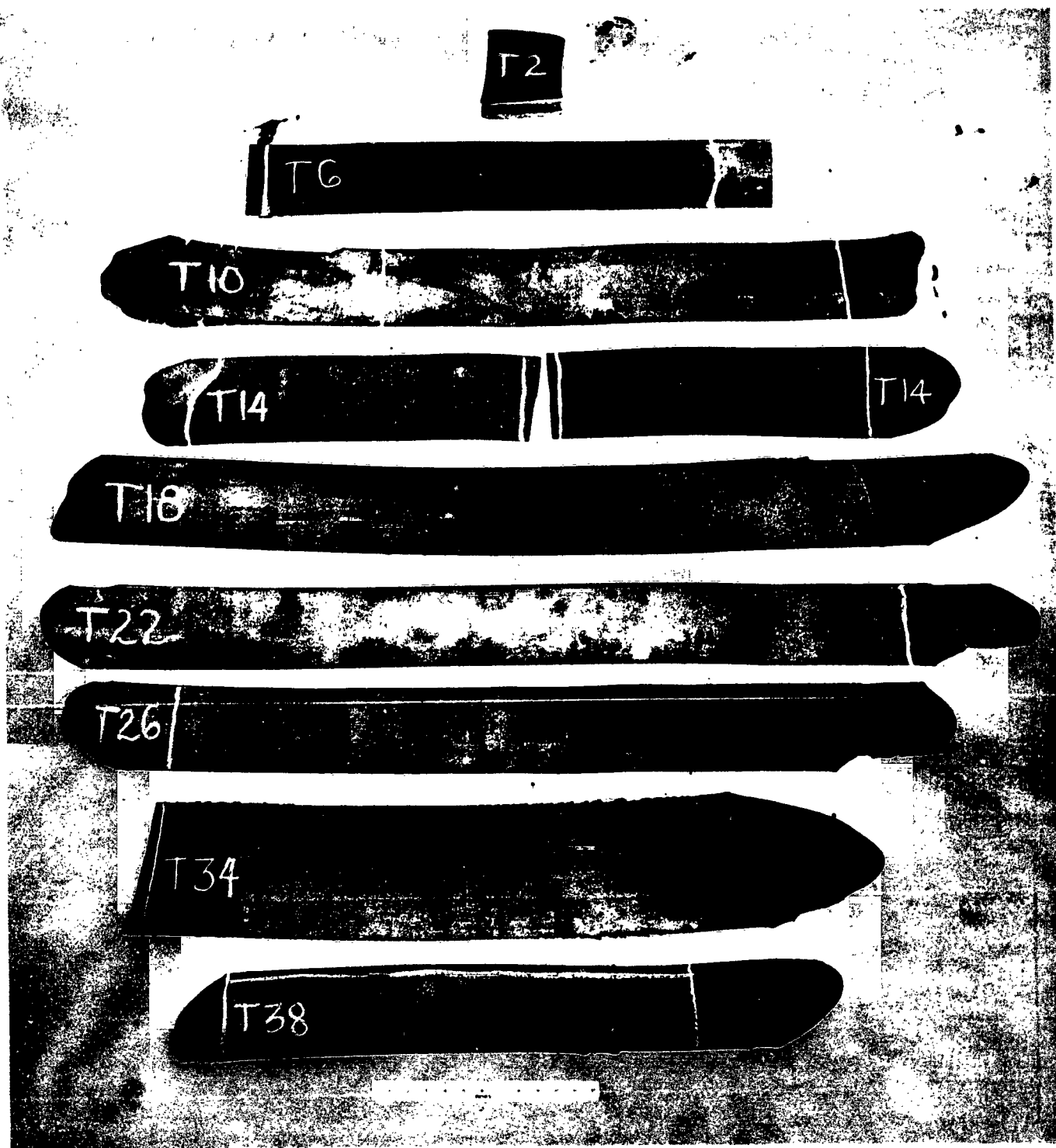


FIGURE 20 - NOMINAL 0.125" MOLD OUT
PRODUCED FROM SHEET BAR ROLLED AT 2800°F.

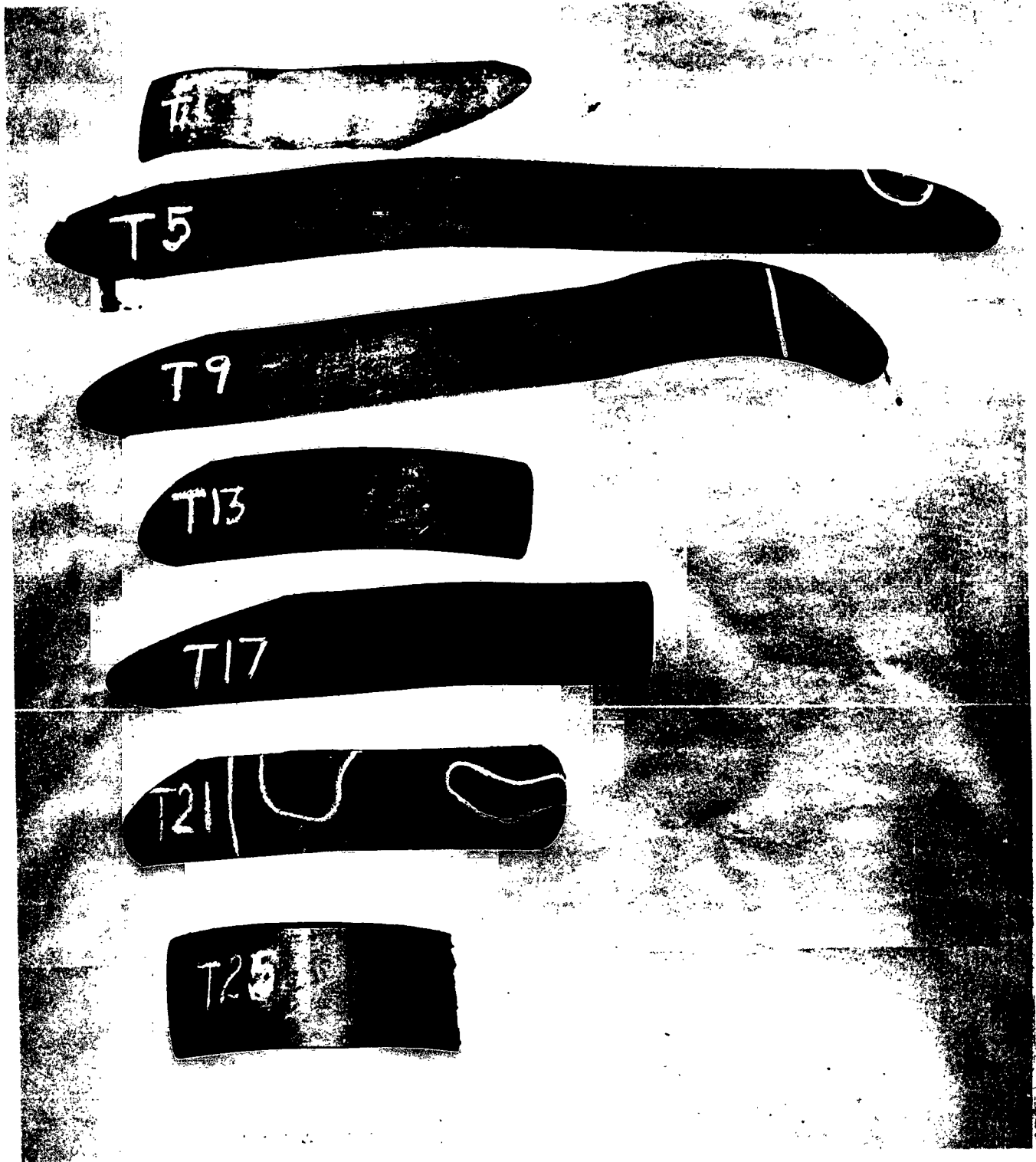


FIGURE 21 - NOMINAL 0.125" MOLD OUT
PRODUCED FROM SHEET BAR ROLLED AT 3200°F.

TABLE V
Visible Contamination Depths in Nominal
0.125" Mold Out

| Mold Out Code | Rolling Temp. °F. | Contamination Recrystallized Layer | Depths Wrought Layer |
|------------------|-------------------------|--|-------------------------|
| T-1 | 3200 | Torn Out | None |
| T-5 | 3200 | .004" | None |
| T-9 | 3200 | .001"-.009" | None |
| T-13 | 3200 | .001"-.008" | None |
| T-17 | 3200 | Torn Out | None |
| T-21 | 3200 | Up to .007" | None |
| T-25 | 3200 | .001"+ | None |
| T-6 | 2800 | None | .001"-.0015" |
| T-10 | 2800 | .001"-.002" | .001" |
| T-14 | 2800 | .001" | .001" |
| T-18 | 2800 | Torn Out | -- |
| T-22 | 2800 | .001"-.0015" | .001"-.0015" |
| T-26 | 2800 | .001" | .001" |
| T-34 | 2800 | .001"-.0015" | .001"-.0015" |
| T-38 | 2800 | .001"-.0015" | .001" |
| T-3 | 2400 | None to .001" | None |
| T-11 | 2400 | None to .001" | None to .001" |
| T-15 | 2400 | None | None |
| T-19 | 2400 | None | None |
| T-23 | 2400 | None | None to .002" |
| T-27 | 2400 | .0005" | .0005" |
| T-35 | 2400 | .0005"-.001" | None |
| T-39 | 2400 | .0005" | .0015" |
| T-8 | 2000 | None | None |
| T-12 | 2000 | None | None |
| T-20 | 2000 | None | None |
| T-24 | 2000 | None | None |
| T-28 | 2000 | None | None |
| T-40 | 2000 | None | None |

possibly because the rolling temperature was high enough to recrystallize any contaminated area. However, the fine recrystallized surface layer (up to .010") attributed to iron contamination would contain any interstitial contaminants present.

Mold out rolled at 2800°F. exhibited both a recrystallized surface layer of up to .002" and below this a .001" to .0015" thick wrought layer. Therefore interstitial contaminants penetrated to a depth of as much as .0035". Mold out rolled at 2400°F. and 2000°F. exhibited very little visual evidence of surface contamination.

Carbon, oxygen, nitrogen and iron analyses are being obtained on selected pieces of mold out. Table VI shows an increase in iron content with rolling temperature from about 0.0019% in the ingot or sheet bar to 0.006% to 0.008% in mold out rolled at 3200°F. Assuming all but .0019% iron is concentrated at the surface, iron contents in the visible surface layer can be calculated at up to 0.1%. Analyses of mold out rolled at 2000°F. and 2400°F. failed to show a significant increase in iron from the ingot analysis of 0.0019%. Analyses of a .005" surface layer pickled from mold out rolled at 3400°F. on Navy Contract NOas 59-6142-c showed an iron content of 7.5% indicating that a sharp increase in iron pick-up occurs with increasing rolling temperatures above the melting point of roll material.

TABLE VI
Effect of Rolling Temperature on
Surface Iron Content of Nominal
0.125" Mold Out

| Mold Out Code | Rolling Temp. °F. | Visible Depth of Contamination Attributed to Iron | Iron Content of Full Mold Out Cross Section | Iron Content of Contaminated Layer (Calculated) |
|---------------------|-------------------------|---|--|---|
| T-1 | 3200 | 0.004" | 0.006% | 0.072% |
| T-5 | 3200 | 0.004" | 0.006% | 0.073% |
| T-13 | 3200 | 0.0047" | 0.008% | 0.110% |
| T-34 | 2800 | 0.0025" | 0.0033% | 0.034% |
| T-3 | 2400 | 0.001" | 0.0015%* | -- |
| T-35 | 2400 | 0.001" | 0.0023%* | -- |
| T-8 | 2000 | 0.0005" | 0.0017%* | -- |

* These results indicate no Iron pick-up
as conditioned sheet bar contained
0.0019% Iron.

3. Hardness

As-rolled hardnesses of all 0.125" mold out are plotted against temperature in Figure 22. Except for a few extreme values, the hardnesses developed at rolling temperatures of 2000°F. and 2400°F. are similar, around 350 to 370 DPH. The greatest decrease in hardness occurs with an increase in rolling temperature from 2400°F. to 2800°F. There is considerable overlap in hardness between rolling temperatures and this is related to sheet bar forging practice. Figure 22 shows that minimum (below the average) hardnesses, at rolling temperatures up to 2800°F., occurred in mold out rolled from heavily hot-cold forged sheet bar while in general, maximum hardnesses occurred in mold out rolled from sheet bar exhibiting hot forged structures. This effect results from the short time 2950° anneal prior to rolling which recrystallized and softened hot-cold forged sheet bar but had a minimal effect on the structure of hot worked or lightly hot-cold worked sheet bar.

Mold out rolled at 3200°F. showed a reversed effect as peak hardnesses of about 300 DPH were developed from hot-cold forged sheet bar while minimum hardnesses of about 260 DPH were developed in mold out rolled from true hot forged sheet bar. Since sheet bars rolled at 3200°F. were not annealed prior to rolling, the higher starting hardness level of hot-cold forged sheet bar was maintained to finished mold out.

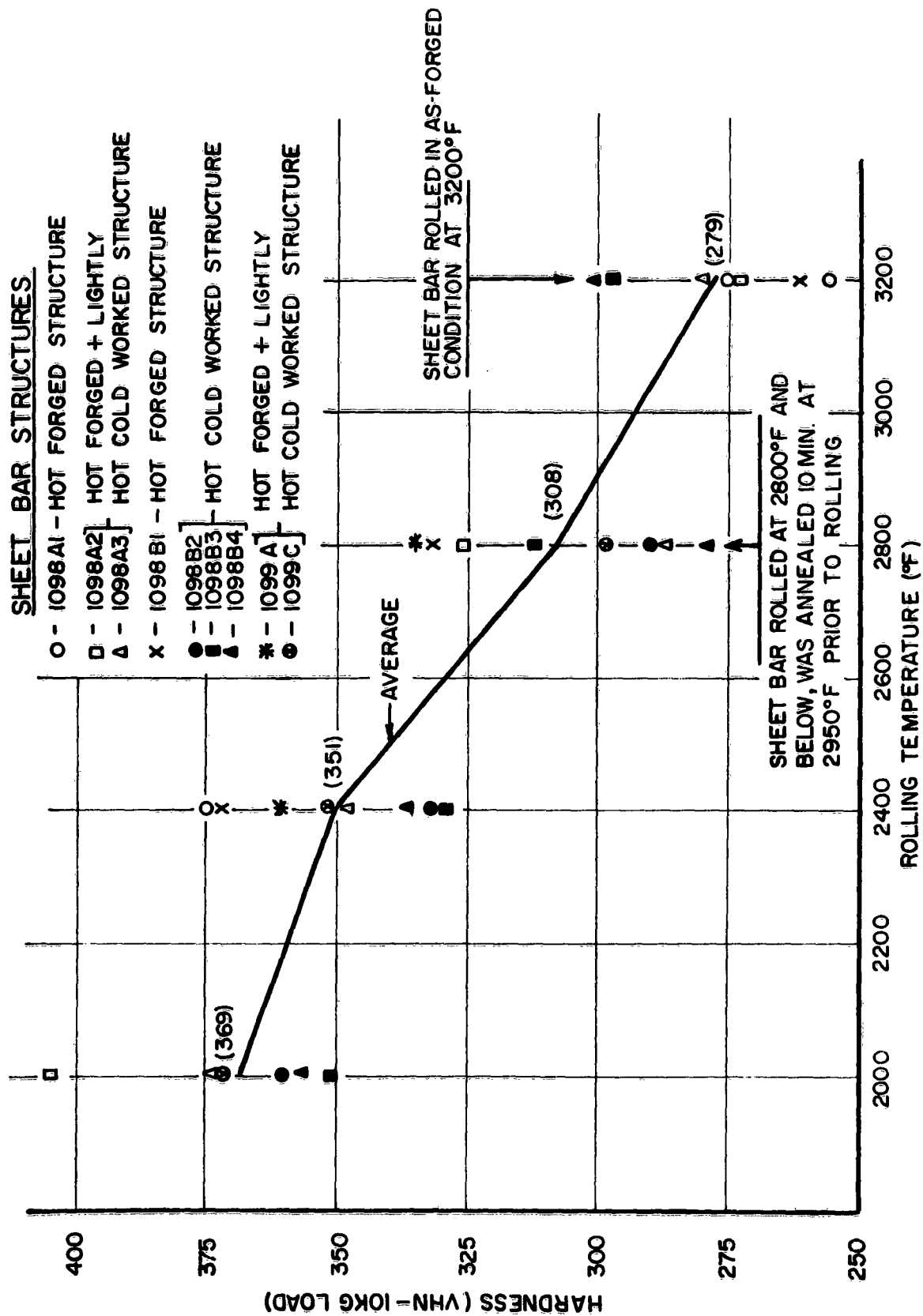


FIGURE 22
AS-ROLLED HARDNESS OF 0.125" MOLD OUT

4. Evaluation Remaining

Mold out evaluation is still in progress. Recrystallization curves are being developed as well as longitudinal and transverse tensile data on as-rolled, as-rolled and conditioned, and conditioned and recrystallized mold out. Surface chemistry data is being obtained on sheet bar and mold out to provide a more quantitative measure of iron or interstitial contamination.

IV. Purity of InFab Atmosphere

Steps have been taken to improve the InFab atmosphere and, in fact, to replace the graphite susceptor induction rolling mill furnace with a resistance heated, tungsten element furnace which will eliminate a prime source of impurities for Phase V rolling.

Results of the effectiveness of the equipment modifications will be reported in the next quarterly report.

V. Summary

Forging and evaluation of sheet bar has completed the Phase III program. Nine of eleven billets were successfully forged to sheet bar and these nine can be grouped according to structure as follows:

- (2) hot forged structures
- (4) hot forged to lightly hot-cold worked structures
- (3) hot-cold forged structures

The most uniform, fine, hot forged structure was produced in extruded billets forged entirely at 3000°F. to 3200°F., however carbides were found almost entirely at the grain boundaries in this piece. Sheet bar forged from cast billets exhibited the most coarse, non-uniform structures with carbides both at the boundaries and within the matrix.

Sheet bar containing only light hot-cold work exhibited recrystallization temperatures above 3000°F. while the hot-cold forged sheet bars all fully recrystallized at 2850°F. to 2900°F. One hour anneals above 2800°F. redistributed the carbides in hot forged sheet bar by reducing the boundary carbide and increasing the amount of fine matrix precipitate.

Twenty-nine of 34 sheet bar sections were successfully rolled to nominal 0.125" intermediate gauge. Sections which failed to roll were from hot-forged sheet bar and exhibited intergranular fractures. Microstructural examination revealed that two types of contamination could occur during mold out rolling. One is attributed to iron, the other to interstitial impurities. Quantitative data has been obtained showing an increase in iron contamination with rolling temperature, but as yet the degree of interstitial contamination has not been determined.

Evaluation of intermediate gauge, including chemistry, hardness, recrystallization and tensile data is in progress. Upon completion of this evaluation, recommendations as to the forging and rolling variables, to be used in the production of the Phase V evaluation sheet, will be made.

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